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IRON OXIDE NANOPARTICLES TO BREAK THE TUBERS DORMANCY OF THE WORLD'S WORST WEED THE CYPERUS ROTUNDUS

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ABSTRACT

Purple nutsedge (*Cyperus rotundus* L.) is a persistent and prolific weed competing with crops for natural resources, results in heavy losses of crops. Phenols present in the tubers control the germination of tubers. Degrading the phenol break open the dormancy and induce the germination of all buds present in the tubers at once. Once tubers are germinated, it can be controlled by either chemical or cultural means results in reduced weed seed bank size. The novel magnetic iron oxide nanoparticles (nano-adsorbent) is quite efficient for degrading phenols present in the *Cyperus rotundus* tubers.A laboratory experiment was carried out at the Department of Nano Science and Technology, Tamil Nadu Agricultural University, Coimbatore to break the dormancy of *Cyperus rotundus* tubers using Iron oxide nanoparticles by degrading the phenols. The Iron oxide nanoparticles were synthesized by chemical precipitation method and characterized by Fourier Transform Infrared Spectroscopy (FTIR), X-Ray powder Diffraction (XDR) and Transmission Electron Microscope (TEM). Iron oxide nanoparticles at the concentration of 3.0 g kg tubers^{-1} recorded higher percentage of phenol degradation i.e 89.0 per cent over control, which was on par with 2.5 g kg tubers^{-1} i.e. 87.1 per cent over control. Advanced oxidation processes (AOP) are widely used for the removal of recalcitrant organic constituents such as phenols. In the case of the AOPs, the generation of hydroxyl radicals takes place through a catalytic mechanism in which the iron oxide nanoparticles play a very important role in phenol degradation. By the way of breaking dormancy factor, the germination percentage of the iron oxide nanoparticles treated *Cyperus rotundus* tubers was increased.

KEYWORDS: Cyperus Rotundus, Dormancy, Germination, Iron Oxide Nanoparticles, Weed

INTRODUCTION

The purple nutsedge (*Cyperus rotundus*) causes serious problems in many crops all over the world than any other weed (Kadir *et al.*, 2000). Often forms dense colonies and greatly reduces the crop yields. It was considered as the one of the world's worst weed (Holm *et al.*, 1977). The sedge weed propagates mainly by producing a complex underground system of rhizomes, basal bulbs and tubers (Stoller, 1975). The tubers remain viable for more than three years and pass over the harsh weather period by preventing the germination of buds present in the tubers. The bud dormancy in tubers has been suspected by many workers as being due to the presence of inhibitors in the tuber. Jangaard *et al.,* (1971) reported that increasing phenolic compounds and abscissic acids in the tubers reduced the sprouting**.** Due to dormancy, the tubers may sustain in the soil for longer time and interfere with the crops raised in the following season. Under this situation, new strategies have to be designed to break the dormancy and killing the sprouted tubers chemically or desiccating the germinated tubers

culturally will helps to reduce the incidence and carry over to the next season. The newly emerging science, the nanotechnological approach throws some light to manage this world's worst weed with the help of nanoparticles (Gu *et al*., 2009). With this background the research work has been programmed to synthesize the iron oxide nanoparticle to degrade the phenols present in the tubers of *Cyperus rotundus.*

Advanced oxidation processes (AOPs) have been realized as efficient technologies for phenol degradation (Bach *et al.,* 2010). In AOPs powerful reactive species like hydroxyl radicals were generated by specific chemical reactions in aqueous solutions. Hydroxyl ions were able to destroy even the most recalcitrant organic molecules such as phenols and convert them into relatively benign and less persistent end products such as CO_2, H_2O and inorganic ions. Incase of phenol degradation, the final product was carbon dioxide and all carbon atoms of phenol were transformed to CO_2 (Barka *et al.,* 2013). Among AOPs processes, heterogeneous photocatalyst using artificial or solar irradiation have been recognized to be effective for the degradation of several types of phenolic compounds (Ahmed *et al.,* 2010).

With the background information, Iron oxide ((Fe_2O_3) nanoparticles were selected as a photocatalyst and synthesized in our laboratory by chemical precipitation method. The Fe_2O_3 nanoparticles are act as nano-adsorbent and one of the potential elements for the degradation of phenol. It is used for oxidative degradation of phenols. The phenol in an aqueous solution removed up to 98 per cent using Fe_2O_3 nanoparticles (Tavallali and Shiri, 2012). The phenol removal was very rapid for first half an hour and attained the equilibrium slowly in 2 hours. 0.3 grams per litre of nano adsorbent used to attain 98 per cent of phenol removal. The removal efficiency of phenol was increased with an increase in Fe_2O_3 nanoparticles dosage and decreased with an increase in initial phenol concentration (Muthukumar, 2012). With this scope, the present study was conducted which paves way for the faster degradation of phenols present in the tubers, could be a promising way to break dormancy and have potential application in weed management.

MATERIALS AND METHODS

Cyperus rotundus tubers were collected from various fields for the analysis. They were sorted based on diameter to maintain uniformity. From this tubers of 3 cm diameter were chosen as samples for phenol degradation and dormancy breaking studies.

The Reagents

Ferric chloride, sodium hydroxide, tween 20, ethanol, sodium carbonate, folin reagent were purchased from Otto and as it is without any further purification.

Synthesis of Iron Oxide (Fe_2o_3) Nanoparticles

0.1 N $FeCl_3$ and 0.3 N NaOH and 1 per cent tween 20 solution was prepared. 50 ml of $FeCl_3$ solution was taken in a 250 ml beaker and it was added with 1 ml of 1 per cent tween 20. This solution was stirred in a magnetic stirrer. 50 ml sodium hydroxide solution was added to the above solution drop by drop. This was stirred well at a temperature of 110^0C until the full solution was consumed. This solution was kept for 2 hrs for settlement of particles. Collected particles were dried and stored (Ozaki *et al.,* 1990).

Phenol Degradation Studies in the *C.rotundus* Using Iron Oxide Nanoparticles

Tubers were treated with Fe_2O_3 nanoparticles at the concentration of 0.5, 1.0, 1.5, 2.0, 2.5 and 3.0 g kg of tubers^{-1}. It was sonicated for 20 minutes and kept undisturbed for 2 hours. 500 mg of treated tuber sample was taken and chopped

into small bits. Then 5 ml ethanol was added and boiled for ten minutes to avoid oxidation of phenols by poly phenol oxidase. After the sample was cooled, macerated with 80 per cent ethanol and final volume was made upto 5 ml. Then it was centrifuged at 10,000 rpm for 10 minutes. The supernatant was collected and volume was made to 10 ml with distilled water.

From the supernatant 1 ml was taken in a test tube for analysis. 1 ml of folin reagent and 2 ml of 20 per cent sodium carbonate solution was added. It was allowed for 30 minutes for colour development at room temperature and OD was measured at 660 nm in uv-vis spectrophotometer. Same procedure was followed for the standards (Pyrocatechol) also. Ran the blank simultaneously.A standard curve was drawn using concentration (X axis) versus absorbance (Y axis) and the concentration of total phenols in the sample was calculated.

Germination Studies of Iron Oxide Nanoparticles Treated *C.rotundus*

The germination percentage of Fe_2O_3 nanoparticles treated *C.rotundus* tubers were studied to assess the dormancy breaking ability of Fe_2O_3 nanoparticles. The tubers were sown in soil media and germination percentage was recorded for each treatment.

RESULTS AND DISCUSSIONS

Characterization of Synthesized Fe_2O_3 Nanoparticles

This is one of the most widely used techniques for structural characterization of iron oxide nanoparticles. The size and shape of the nanoparticles, preparation conditions, addition of support materials and the capping agent are major factors in catalysis. The image (Figure.1) shows FTIR spectra of Fe_2O_3 nanoparticles. Infrared studies were carried out in order to ascertain the purity and nature of the metal nanoparticles. The peak at 3441 cm^{-1} has been assigned to the C-H stretching. The strong absorption band at 470 cm^{-1} corresponding to the Fe_2O_3 nanoparticles (Awwad and Salem, 2012).

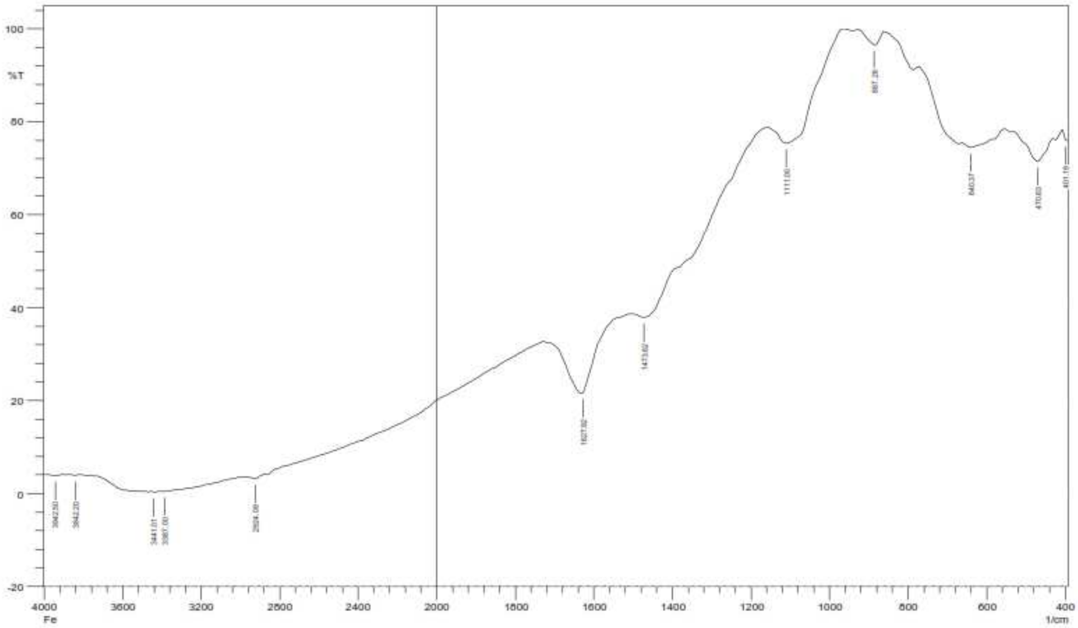

Figure 1: FTIR Spectra of Fe_2O_3 Nanoparticles

X- ray diffraction pattern of Fe_2O_3 nanoparticles is depicted in the Figure. 2. The characteristic peak at an angle of (2θ) 31.9° was observed. The results conforms the JCPDS card no. 40-1139. The crystal unit cell of the nanoparticles was found to be hexagonal.

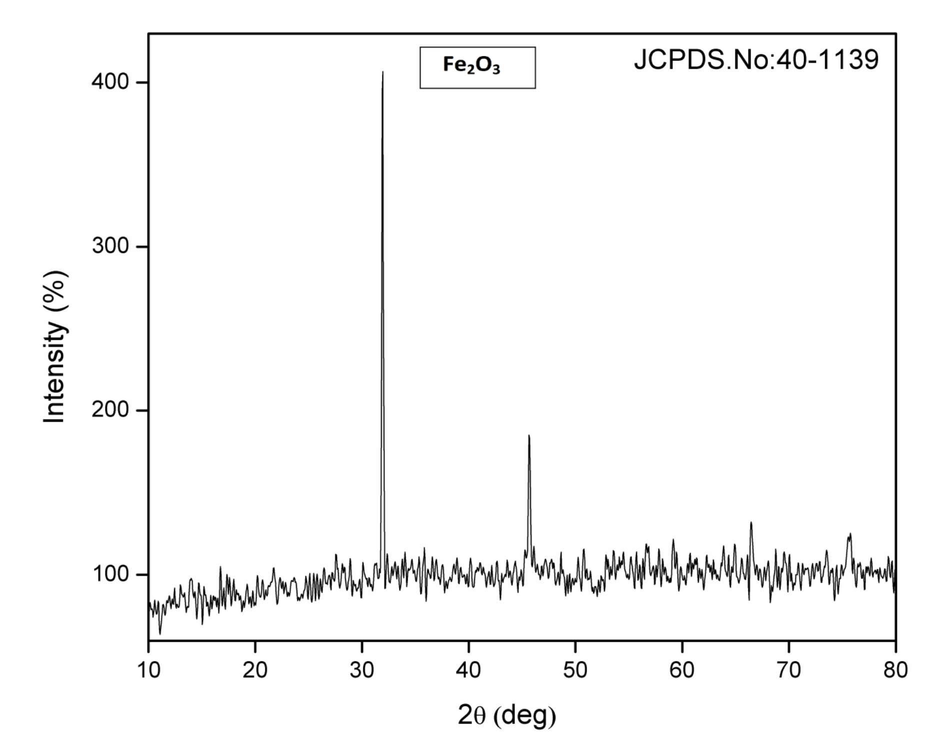

Figure 2: XRD Pattern of Fe_2O_3 Nanoparticles

The image (Figure. 3) obtained from TEM for Fe_2O_3 oxide nanoparticles showed that, they are spherical in shape with a smooth surface morphology. The diameter of the nanoparticles is found to be approximately 40.4 nm.

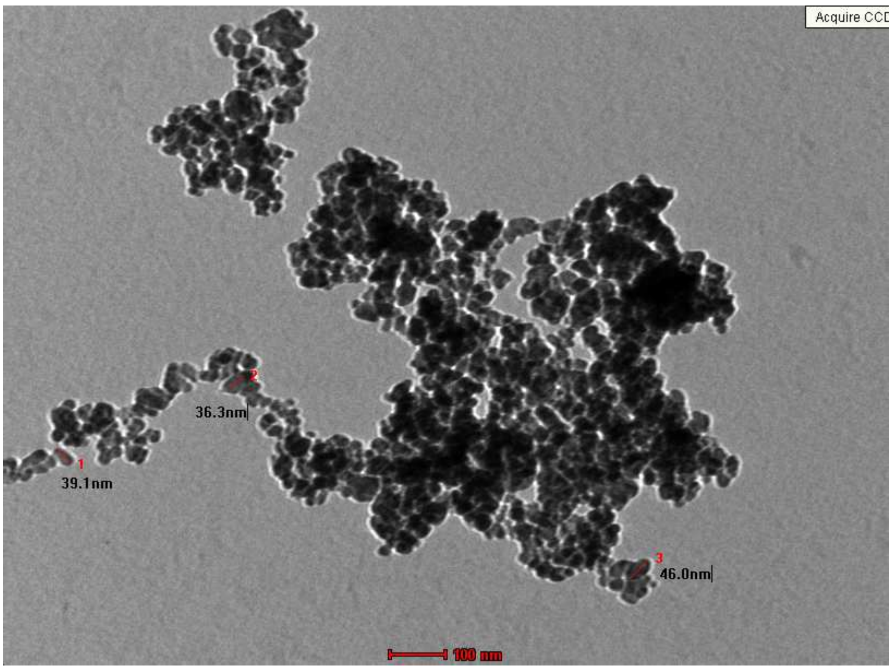

Figure 3: TEM Image of Iron Oxide Nanoparticles

Phenol Degradation of *C.rotundus* Using Fe_2O_3 Nanoparticles

Degradation of phenols present in *Cyperus rotundus* tubers by Fe_2O_3 nanoparticles was presented in Table 1. The dosage of Fe_2O_3 nanoparticles significantly influenced the degradation efficiency of phenol. Fe_2O_3 nanoparticles at the concentration of 3.0 g kg tubers^{-1} (T_7) recorded higher percentage of phenol degradation i.e 89.0 per cent over control, which was on par with T_6 (2.5 g kg tubers^{-1}) i.e. 87.1 per cent over control. Advanced oxidation processes are widely used for the removal of recalcitrant organic constituents such as phenols. These procedures are based on generating highly oxidative HO radicals in the reaction medium (Lazar *et al.,* 2012). The oxidation process being determined by the very high oxidative potential of the HO radicals generated into the reaction medium by different mechanisms (Pera-Titus *et al.*, 2004).

A complete mineralization of the phenolic compounds is not necessary, being more worthwhile to transform them into biodegradable aliphatic carboxylic acids followed by a biological process (Wang and Wang, 2007). In the case of the AOPs Fenton type procedure (hydrogen peroxide and Fe_2O_3 nanoparticles as catalyst), the generation of hydroxyl radicals takes place through a catalytic mechanism in which the Fe_2O_3 nanoparticles play a very important role (Andreozzi *et al.*, 1999; Esplugas, *et al.*, 2002)

Germination Studies of Fe_2O_3 Nanoparticles Treated Tubers

Germination percentage of Fe_2O_3 nanoparticles treated *C,rotundus* tubers was depicted in Fig. 4. Since, the dosage of Fe_2O_3 nanoparticles significantly influenced the degradation efficiency of phenol; it has influence on germination percentage too. Fe_2O_3 nanoparticles at the concentration of 3.0 g kg tubers^{-1} (T_7) recorded higher germination percentage i.e 61.3 percentage.It was followed by Fe_2O_3 nanoparticles at 2.5 g kg tubers^{-1} gives 53.3 percentage germination. The untreated control recorded only 13.3 percentage germination. The bud dormancy in tubers is due to the presence of inhibitors in the tuber. Jangaard *et al.,* (1971) reported that increasing phenolic compounds and abscissic acids in the tuber may inhibit sprouting. Due to the production of hydroxyl radicals by Fe_2O_3 nanoparticles, the phenols present in the tubers were degraded by the AOPs. Hence the germination percentage of the treated tubers was increased with the increased Fe_2O_3 nanoparticles dosage, by breaking the germination inhibitor.

Table 1: Effect of Iron Oxide (Fe_2O_3) Nanoparticles in Degradation of Total Phenols of *Cyperus rotundus* Tuber

S. No	Concentration of Fe_2o_3 NPS (G Kg of Tuber^{-1})	Phenol Concentration (Mg G of Tuber^{-1})
1	T_1 - Control	21.78
2	T_2 - 0.5	10.67
3	T_3 - 1.0	6.39
4	T_4 - 1.5	5.70
5	T_5 - 2.0	4.41
6	T_6 - 2.5	2.79
7	T_7 - 3.0	2.38
SEd		**0.42**
CD (P=0.05)		**0.91**

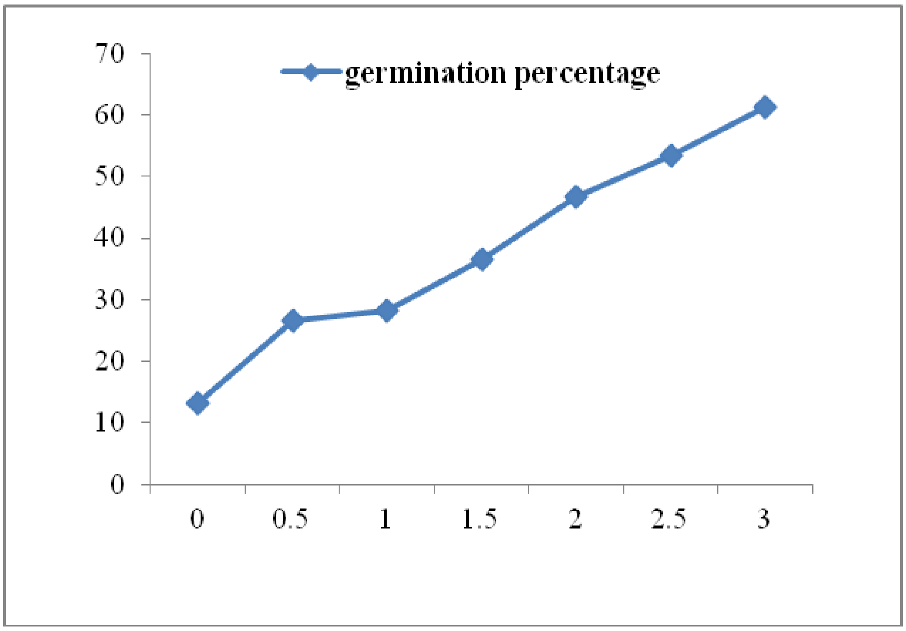

Figure 4: Effect of Fe_2O_3 Nanoparticles in Germination Percentage of *Cyperus rotundus* Tuber

CONCLUSIONS

Iron oxide nanoparticles at the concentration of 3.0 g kg tubers^{-1} (T_7) recorded higher percentage of phenol degradation i.e 89.0 per cent over control, which was on par with T_6 (2.5 g kg tubers^{-1}) i.e. 87.1 per cent over control. Advanced oxidation processes (AOP) are widely used for the removal of recalcitrant organic constituents such as phenols. In the case of the AOPs, the generation of hydroxyl radicals takes place through a catalytic mechanism in which the iron oxide nanoparticles play a very important role in phenol degradation. By the way of breaking dormancy factor, the germination percentage of the iron oxide nanoparticles treated *Cyperus rotundus* tubers was increased, which has a potential application in weed management.

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